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(54) Modified soda lime product

(57) A process for the manufacture of a soda lime product comprises the steps of:

- (a) forming an aqueous slurry containing calcium hydroxide and a gel of an inorganic material by precipitating calcium hydroxide from an aqueous solution of a water-soluble calcium salt by the addition of an aqueous alkaline hydroxide solution in the presence of or with co-precipitation of the inorganic gel;
- (b) separating calcium hydroxide and inorganic gel from the slurry;
- (c) heating the separated calcium hydroxide and inorganic gel, together with sodium hydroxide and/or potassium hydroxide, under super atmospheric pressure; and
- (d) adjusting the water content of the heated mixture from (c) to the desired level to give an absorbent soda lime material.

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SPECIFICATION

Soda lime

- 5 This invention is concerned with improvements in and relating to the manufacture of soda lime.

Soda lime is a well known absorbent for carbon dioxide and is used for that purpose in, for example, breathing and anaesthetic apparatus. Soda lime is also used in the refining or purification of petroleum hydrocarbons to remove sulphur-containing impurities.

Soda lime basically comprises a solid mixture of calcium hydroxide with a minor proportion by weight of sodium hydroxide and/or potassium hydroxide, the mixture also containing some water (typically up to 21% by weight) to "activate" the product, that is to give it the desired absorbent properties. It is commonly prepared by a process comprising the steps of slaking lime (CaO), or mixing hydrated lime, Ca(OH)_2 , with aqueous sodium hydroxide and/or potassium hydroxide solution to produce a pasty mass, drying the mass (generally after sub-division thereof) and finally (typically after crushing and classifying the dried material) adding some water to the dried material to activate it. In theory the steps of drying and subsequent addition of water could be replaced by a simple drying step to give the desired water content but in practice this is not preferred.

The product obtained by the above process is undoubtedly useful but does suffer from disadvantages in that whilst an effective absorbent for carbon dioxide it exhibits, in many practical applications, only about 50% of the theoretically expected absorbency. Further, soda lime so obtained may be insufficiently hard for some purposes in that it may be abraded to give a dust which is itself an irritant and may also tend to clog beds packed with granular soda lime. Further, when soda lime granules are produced by a process involving crushing and classification of a dried soda lime material, there may be undesirable wastage in the form of undersized particles or dust.

It is an object of the present invention to provide a process for the manufacture of an improved soda lime product.

According to the invention there is provided a process for the manufacture of a soda lime product which comprises the steps of:

- (a) forming an aqueous slurry containing calcium hydroxide and a gel (a fluid colloidal dispersion) of an inorganic material by precipitating calcium hydroxide from an aqueous solution of a water soluble calcium salt by the addition of an aqueous alkaline hydroxide solution in the presence of or with co-precipitation of the inorganic gel;
- (b) separating calcium hydroxide and inorganic gel from the slurry;

(c) heating the separated calcium hydroxide and inorganic gel, together with sodium hydroxide and/or potassium hydroxide under super-atmospheric pressure; and

- 70 (d) adjusting the water content of the heated mixture from step (c) to the desired level to give an absorbent soda lime material

In the first stage of the process of the invention, calcium hydroxide is precipitated from an aqueous alkaline of a salt thereof by the addition of aqueous alkaline hydroxide solution, especially a solution of sodium hydroxide and/or potassium hydroxide. The precipitation is effected in the presence of, or with the co-formation of an inorganic gel. Suitable gels include silicate gels and alumina gels each of which may be produced by adjusting the pH of a solution of an appropriate precursor thereof, e.g. sodium silicate in the case of a silicate gel and aluminium nitrate in the case of an alumina gel. Suitable water-soluble calcium salts for use in the process of the invention include calcium chloride and calcium nitrate. We have found that aqueous solutions of commercially available water-soluble calcium salts, such as calcium chloride and are in themselves effective pH adjusters to induce formation of a silicate from a sodium gel, from a sodium silicate solution. In the case of alumina gels, however, we have found that gel formation is effectively induced by the addition of an alkaline hydroxide solution (for simultaneous precipitation of calcium hydroxide). We have further found, in general, that silicate gels give better overall results and, hence, it is preferred to carry step (a) by the steps of:

- (i) adding an aqueous solution of calcium chloride to an aqueous solution of sodium silicate to form a silicate gel therein;
- 105 (ii) adding an aqueous alkaline hydroxide solution to the silicate gel-containing solution to precipitate calcium hydroxide therein.

Where, however, an alumina gel is to be employed, step (a) is suitably carried by the steps of

- (i) forming a mixed aqueous solution of calcium nitrate and aluminium nitrate; and
- (ii) adding an aqueous alkaline hydroxide solution to the mixed solution to simultaneously precipitate out calcium hydroxide and to form an alumina gel.

In any event, the weight ratio of calcium salt (calculated as calcium hydroxide) to inorganic gel is suitably from 1:1 to 15:1, preferably from 2:1 to 8:1.

The alkaline hydroxide should be added at least an amount sufficient to precipitate out all the calcium as calcium hydroxide. The slurry produced in step (a) will also contain dissolved components, especially the salt of the cation of the alkali hydroxide and the anion of the water-soluble calcium salt (hereinafter simply referred to as by-product salt) and any excess alkaline hydroxide.

130 In the second stage of the process, step

(b), the solid components of the slurry are separated by any suitable solid-liquid separation method such as filtration, centrifugation or decantation. The separated solids are then

5 preferably washed with water to remove the bulk of water-soluble components. As will be appreciated, if the alkaline hydroxide employed in step (a) is sodium hydroxide or potassium hydroxide, this in itself can serve as all or part
10 of the sodium hydroxide or potassium hydroxide component of the final soda lime product. In this case, accordingly, the washing is preferably not taken to completion since it is desirable that some alkali metal hydroxide remain. As a general rule the washed product
15 should contain from 0.5 to 10%, preferably from 1.5 to 3.0%, by weight of sodium or potassium hydroxide, based on the weight of calcium hydroxide. It has been found that the presence of residual small amounts of by-product salts do not adversely affect the overall performance of the final product. Should too much sodium or potassium hydroxide be removed in the washing step, additional sodium
25 or potassium hydroxide can be added (as solid or as a concentrated aqueous solution) to the washed product before the succeeding stage, namely step (c) in which the washed calcium hydroxide/alkali metal hydroxide/inorganic gel product is heated under pressure or auto-
30 claved. Autoclaving is suitably carried out at a temperature from 40 to 200°C, preferably 80 to 140°C, under a pressure greater than 0.7 Bars, preferably greater than 1.5 Bars. The period of autoclaving is suitably from 2 to 24 hours preferably 4 to 12 hours.

After autoclaving, the water content of the product is adjusted to that desired from the final product. This will most conveniently be
40 achieved by the usual steps of drying followed by the addition of the required amount of water. In this case, the product is dried suitably by heating at a temperature of from 40 to 200°C, preferably 80 to 140°C for a
45 period of 4 to 60 hours, preferably 6 to 18 hours. The dried product from the drying step should then be dampened (activated) to give a moisture content of from upto 21% by weight.

50 The product of the drying step is in powder form and thus is generally not a suitable form for the great majority of the applications of soda lime. Accordingly, before moistening, the product is preferably formed into larger particles, e.g by a granulation or tableting step which may be effected in any suitable manner.

The process, and product, of the invention have a number of advantages over those of the conventional prior art. Thus, the amount
60 of carbon dioxide which may be absorbed by a product in accordance with the invention may be upto 80% of the theoretical and this is believed to be due to the fact that the product of the invention is more porous (i.e.
65 has a greater effective surface area per gram)

and has a better pore size distribution than the product of the prior art. Further, there is much reduced wastage in the process of the invention in that oversize or undersized material formed in the production of dried particulate material can be reused. Depending upon the particular conditions under which the dried product is converted to particulate form harder or more abrasion resistance products (products less liable to form dust) can be obtained.

In order that the invention may be well understood the following Example is given by way of illustration only.

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EXAMPLE

500 ml of an aqueous calcium chloride solution (157g CaCl_2) were added to 500 ml of an alkaline aqueous silica solution (21g SiO_2) in a
85 stirred reactor. A white gel was formed and there were then added to the reactor 500 ml of aqueous hydroxide solution (106g NaOH) whereupon calcium hydroxide was precipitated to form a gelatinous precipitate. The precipitate was washed and filtered (2000 ml) de-ionised water) and then charged to a glass pressure reactor in which it was autoclaved at
90 120°C at 2.3 bar for 8 hours. The autoclaved material was then dried in an reactor over at
95 120°C for 17 hours.

The resultant powder, on wetting with water, had the following characteristics:-

BET surface area	90 m ² /g
100 (CO ₂ absorption/USP)	36 wt.%

CLAIMS

1. A process for the manufacture of a soda lime product which comprises the steps of:

(a) forming an aqueous slurry containing calcium hydroxide and a gel of an inorganic material by precipitating calcium hydroxide from an aqueous solution of a water-soluble calcium salt by the addition of an aqueous alkaline hydroxide solution in the presence of or with co-precipitation of the inorganic gel;

(b) separating calcium hydroxide and inorganic gel from the slurry;

(c) heating the separated calcium hydroxide and inorganic gel, together with sodium hydroxide and/or potassium hydroxide, under atmospheric pressure; and

(d) adjusting the water content of the heated mixture from (c) to the desired level to give an absorbent soda lime material.

2. A process as claimed in claim 1 in which step (a) is carried out by:

(i) adding an aqueous solution of calcium chloride to an aqueous solution of sodium silicate to form a silicate gel therein; and

(ii) adding an aqueous alkaline hydroxide solution to the silicate gel-containing solution to precipitate calcium hydroxide therein.

3. A process as claimed in Claim 1 substantially as hereinbefore described with reference to the Examples.

4. A soda lime product when obtained by
5 a process as claimed in any one of the preceding claims.

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